# Solid-phase extraction for oil and grease analysis

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**ABSTRACT:** Conventional oil and grease analysis that involves liquid-liquid extraction (LLE) has many disadvantages, which include poor reproducibility, emulsion formation, large solvent usage, and loss of volatile and semivolatile compounds during evaporation of the solvent. Therefore, an alternative method using octadecyl siloxane (C18) solid-phase extraction (SPE) columns was developed to overcome these analytical problems. The amount of the solvent was reduced and more reproducible results were obtained using this C18 SPE method. The time required for analysis is approximately the same for both methods. Higher recovery of semivolatile compounds also was obtained. The proposed C18 SPE method was also found to be comparable to those obtained from commercially available columns. *Water Environ. Res.*, **69,** 368 (1997).

**KEYWORDS:** analysis, extraction, grease, oil, stormwater.

According to *Standard Methods* (APHA, 1995), oil and grease is defined as ''any material or substance that is soluble in the solvent.'' It does not measure the presence of any specific compound but is an important analytical procedure for environmental samples. The conventional liquid-liquid extraction (LLE) procedure for oil and grease analysis is plagued by various analytical problems such as poor or inconsistent recovery, emulsion formation, usage of large solvent volume, and loss of volatile and semivolatile compounds during evaporation of the solvent. Stenstrom *et al.* (1986) reviewed the development of oil and grease analytical procedures and their disadvantages. An alternative method for the oil and grease analysis is needed to overcome these analytical problems and, more importantly, to avoid or reduce the use of solvents that may be greenhouse or smog-forming gases. Solid phase extraction (SPE) is one candidate procedure and has been used extensively during the past 20 years for sample preparation in the analysis of semi- and nonvolatile organic compounds for both environmental samples and for drugs in the pharmaceutical industry. The advantages of using solid-phase extraction are reduced analysis time, cost and labor, and freedom from emulsion formation. Solvent usage is also much less.

The development in recent years of disposable columns with prepacked bonded silica has encouraged the use of solid-phase extraction for environmental and pharmaceutical applications. The most commonly used silica bonded adsorbents include octadecyl (C18), octyl (C8), ethyl (C2), cyclohexyl (CH), diol (OH) and cyanopropyl (CN). Two major uses of the SPE method are sample cleanup and concentration. Sample cleanup is required when impurities in the sample matrix interfere with analyte measurement in the analytical method of choice, such as gas chromatography. Increasing the concentration of analyte is important when the sample is too dilute for direct measurement.

The major obstacle when using the SPE procedure is the method development, where efficiency and precision depend upon the type of analyte, sample matrix, type of sorbents, and elution solvent. General method development for the SPE procedure has been discussed in detail by Chladek and Marano (1984), McDowall *et al.* (1986), and Wells and Michael (1987).

Recently, Analytichem, a division of Varian, developed the EnvirElut™ Oil and Grease column for analysis of oil and grease. 3M (St. Paul, Minn.) also developed a specific type of SPE disk, Empore™ extraction disk, for oil and grease analysis. Both of these proprietary methods have the reported advantage of reduced solvent usage and may have some of the other advantages over liquid-liquid extraction. Unfortunately, not all of the details about the sorbent composition are published. This research was initiated to develop an SPE procedure using commercially available and described materials that have the aforementioned advantages. The goals of this method development are to provide more reproducible results, improve recovery of semivolatile compounds, reduce solvent volume, and reduce analysis time.

## Experimental Procedures

**Instrumentation.** A Sartorius Model 1712MP8 (Brinkmann Instrument Co., Westbury, N.Y.) analytical balance was used for the gravimetric analysis of the recoverable oil and grease. A Masterflex peristaltic pump (Cole-Parmer, Niles, Ill.) was used for the solid-phase extraction procedures.

**Materials and chemicals.** The SPE columns used in this study were 1 000 mg size Mega Bond Elut™ columns (*i.e.*, ethyl [C2], octyl [C8], and octadecyl [C18] siloxane bonded to silica columns) and 500 mg silica gel column obtained from Analytichem (Harbor City, Calif.). Reagent-grade methylene chloride, n-hexane, isopropanol, and concentrated hydrochloric acid used in the SPE procedures were obtained from Fisher Scientific (Tustin, Calif.).

**Sample preparation.** Automobile crankcase oil was used to prepare the working standard solutions for the oil and grease analysis in this study. A stock solution of motor oil was prepared by mixing a known amount of motor oil in 100 mL deionized water using a wrist action shaker (Burrell Scientific, Pittsburgh, Penn.). This sample was used because we wanted to simulate the oil and grease found in urban runoff (stormwater), and vehicle crankcase emissions are known to be large contributors to stormwater pollution (Stenstrom *et al.*, 1984).

**Solid-phase extraction procedures.** Figure 1 shows the setup of the solid-phase extraction. The 1 000-mg C18 column was first conditioned with 5 mL isopropanol followed by 5 mL deionized water. A 500-mL sample was treated by adding 25 mL isopropanol and 1 mL concentrated HCl acid. The sample was then passed through the column at a flow rate of 5 mL/min.

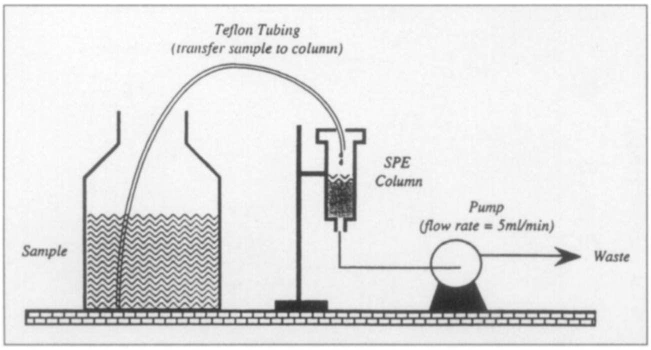

**Figure 1—Experimental setup for the C18 solid-phase extraction.**

To remove oil and grease from the wall of sample container, 5 mL of isopropanol was added into the empty sample container and used to rinse the wall of the container. One hundred millilitres of deionized water containing 0.1% concentrated HCl then was added to the same empty container, and the mixture was passed through the column as before. The column was then dried for ~25 min under vacuum (~44.5 cm Hg).

A tared collection tube was placed under the column after it was dried. The column was eluted with 3 mL of methylene chloride followed by 2 mL of hexane. Each elution fraction in the collection tube was evaporated to dryness at ~55°C under a slow stream of nitrogen gas. The tube then was weighed to determine the mass of oil and grease eluted from the C18 column. The concentration of recoverable oil and grease was determined as follows:

$$\text{Concentration (mg/L)} = \frac{\text{mass of oil and grease eluted (mg)}}{\text{sample volume (L)}} \qquad (1)$$

The mass of oil and grease eluted in Equation 1 is the combined mass of oil and grease eluted in the methylene chloride and hexane fractions.

**Liquid-liquid extraction procedures.** This liquid-liquid extraction (LLE) procedure described in *Standard Methods* (APHA, 1995) (Method 5520B) was used in this study. The extracting solvent used in the LLE was methylene chloride. Methylene chloride frequently is used in research applications instead of Freon® 113 because of the desire to minimize Freon® usage as well as to maximize recovery. The sample volume used in the LLE was 500 mL instead of 1 000 mL as suggested by *Standard Methods.* The sample was acidified to ≤pH 2 using concentrated HCl and then transferred to a separatory funnel. The sample container was rinsed with 15 mL methylene chloride and then added into the separatory funnel. After shaking the funnel vigorously for ~2 min, the funnel was left to stand for 5–10 min until stable layers were formed. The methylene chloride layer then was drained through a funnel that contained a solvent-moistened filter paper into a clean, tared distilling flask. If an emulsion preventing the formation of a clear solvent layer formed, 1 g of sodium sulfate was added to the filter paper cone. The sample then was extracted twice more with 15 mL methylene chloride. The extracts were combined and the filter paper was washed with an additional 5–10 mL of methylene chloride. The solvent was then evaporated at ~55°C under a slow stream of nitrogen gas. The dried flask then was cooled in a desiccator for 30 min and then weighed. A total of 45 mL methylene chloride was used in this procedure. The percentage recovery obtained from the LLE then was compared with those obtained from the modified C18 SPE procedures.

## Results and Discussion

The experimental program evaluated all the major parameters affecting the SPE procedure, except sample flow rate. The impact of C2, C8, and C18 sorbents, sample volume, isopropyl alcohol volume, and oil and grease concentration were all evaluated. Finally, a comparison with LLE and other SPE methods was made.

**Sorbents.** The first step in developing an SPE method is the selection of an appropriate sorbent that will extract oil and grease most efficiently. The recovery of oil and grease from three different sorbents, C2, C8, and C18, was studied and results are shown in Table 1. Among these three sorbents, C18 exhibited the best recovery of oil and grease, with an average percentage recovery of 89%. Table 1 also shows the confidence interval at $\alpha = 0.10$ for the percentage recoveries. The extraction efficiencies of C2 and C8 columns are not significantly different. However, the extraction efficiency of the C18 column was significantly better than both C2 and C8 columns and showed reduced variability in recovery. Thus, C18 column was used for the subsequent extraction of oil and grease.

**Elution volume effect.** Methylene chloride and hexane have been used widely in the extraction of nonpolar compounds. Preliminary C18 SPE studies had demonstrated the efficiencies of these two solvents in eluting oil and grease from the C18 column. In addition to methylene chloride, hexane was also used to elute the oil and grease from the C18 sorbent, and preliminary studies showed that high-molecular-weight hydrocarbons (such as C30 and C33 hydrocarbons) would only be desorbed from the C18 sorbent by hexane. After the appropriate elution solvents were selected, the effect of elution volume was studied so that the optimum elution volume could be determined. The optimum volume is the minimum volume that will elute all the adsorbed organics. Figure 2 shows the obtained percentage recovery of the oil and grease at four different elution volumes. The results show no improvement of extraction efficiencies after the addition of the third elution volume (E3). Therefore, the selected elution volumes used for the C18 SPE are as follows: 3.0 mL of methylene chloride and 2.0 mL of hexane (*i.e.,* E4). The last elution step (*i.e.,* the second 1.0 mL of hexane) is not needed for this application; however, it may

**Table 1—Comparison of percentage recovery of oil and grease using different sorbent.**

| Sorbent (1 000 mg) | Mean ± SD[a] | CL ($\alpha$ = 0.10) |
|---|---|---|
| C2 | 81 ± 8 | 76–86 |
| C8 | 84 ± 4 | 81–87 |
| C18 | 89 ± 2 | 88–90 |

[a] Based on eight replicate extractions of 500-mL sample with prior addition of 25 mL isopropanol to the sample.

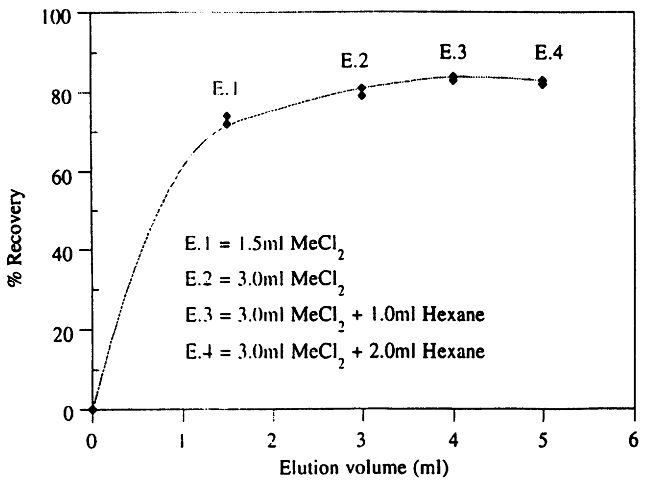

**Figure 2—Effect of elution volume on recovery of oil and grease.**

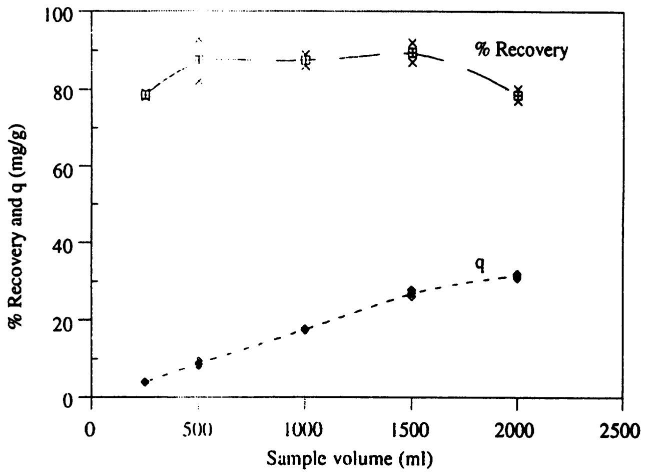

**Figure 3—Effect of sample volume on the recovery of oil and grease.**

be used to insure that all sorbed material is eluted. Other investigators may want to evaluate its use for their applications.

**Isopropanol volume effect.** Sample pretreatment, such as the addition of an appropriate solvent, is known to improve the efficiency of extraction. By adding the solvent into the sample before extraction, the solubility of the least soluble compounds can be increased and thus minimize the physical losses in the sample container. In addition, the solvent also promotes the interaction between C18 bonded phase with the water sample and thus helps to maintain the equilibrium between the solid and liquid phase (McDowall *et al.,* 1986; Chladek and Marano, 1984). The solvent used in this sample pretreatment step is usually the same as the solvent used to condition the SPE column. Therefore, isopropanol (IPA) was added into the sample prior to its passing through the C18 column. Table 2 shows the average percentage recovery of oil and grease using three different IPA volumes.

Isopropanol volumes of 10, 25, and 50 mL were added to 500-mL samples that were subsequently analyzed using the SPE procedure. The recovery using 25 mL of IPA was significantly better ($\alpha = 0.10$) compared with the recovery when using 10 and 50 mL IPA volumes. Therefore, ≥5% (v/v) concentration of IPA is needed to achieve desirable recovery of oil and grease; less than 5% (v/v) of IPA may not be sufficient in promoting the desired interaction between the sorbent with oil and grease compounds in the aqueous sample. Using >5% (v/v) of IPA reduced recovery and may have caused the breakthrough of the oil and grease compounds from the C18 sorbent.

**Table 2—Isopropanol volume effect on the percentage recovery of oil and grease.**

| IPA volume, mL | IPA, % (v/v) | Mean ± SD[a] | CL ($\alpha$ = 0.10) |
|---|---|---|---|
| 10 | 2 | 80 ± 3 | 77–83 |
| 25 | 5 | 89 ± 2 | 87–91 |
| 50 | 10 | 82 ± 1 | 81–83 |

[a] Based on three replicate extractions using 1 000 mg C18 column and 500-mL sample volume.

Oil and grease adsorbs to glass and plastic, and for this reason polytetrafluoroethylene (Teflon™) generally is required to handle any water sample for oil and grease analysis. This usually creates additional cost, and it is often not possible to use polytetrafluoroethylene for all applications. To overcome the adsorption problem of oil and grease to the wall of the glass sample container, a small volume of IPA (5 mL) was added to the sample container after the whole sample had passed through the C18 column. The sample container was then swirled in a circular motion with the added IPA. Then, 100 mL deionized water was added into the same sample container and mixed well with IPA. The IPA and deionized water mixture then were passed through the C18 column. The additional IPA at the end of sample extraction redissolved the oil and grease material from the glass wall of sample container, thus improving the recoveries of extraction. Under similar extraction conditions, it was found that the percentage recovery of the oil and grease, without addition of IPA in the final washing step, was <60%, suggesting that the final IPA wash increased recovery by 20–25%. The greater the concentration of the oil and grease, the more important this washing step becomes.

The use of IPA to improve recovery and reduce oil and grease retention on glassware introduces questions about waste production. Introducing large amounts of solvents into wastewaters from laboratories, especially a production laboratory where large numbers of analyses are performed, is undesirable. Fortunately, IPA is not a listed hazardous air pollutant (Kao, 1994). It is easily biodegradable and has reduced smog (ozone) formation potential compared to many other solvents, such as hexane (Carter, 1994). Its short lifetime in the atmosphere is also low enough to prevent it from becoming a greenhouse gas.

**Sample volume effect.** The mass of oil and grease adsorbed on the C18 SPE column is dependent on the volume of the sample used for the extraction: the greater the sample volume used, the greater the mass of oil and grease transferred to the sorbent. Figure 3 shows the percentage recovery of the oil and grease from five different sample volumes. Each sample volume had similar oil and grease concentration. The results show that

**Table 3—Comparison of percentage recovery of synthetic and environmental spiked samples.**

| Sample | Number of samples | Mean ± SD | CL ($\alpha$ = 0.10) |
|---|---|---|---|
| Synthetic spiked sample | 8 | 89 ± 2 | 88-90 |
| Environmental spiked sample | 10 | 88 ± 4 | 86-90 |

the recovery of oil and grease remained almost unchanged when the volume of the sample increased from 500 to 1 500 mL. It was observed that breakthrough occurs when >1 500 mL sample passed through the 1 000-mg C18 sorbent, as indicated by the decrease of the percentage recovery from ~90% at 1 500 mL to 79% at 2 000 mL. The adsorption capacity ($q$) of the 1 000-mg C18 column was determined as follows:

$$q\ (\text{mg/g}) = \frac{\text{mass of oil and grease eluted (mg)}}{\text{mass of C18 sorbent (g)}} \qquad (2)$$

The maximum capacity of the 1 000-mg C18 SPE column was found to be ~27 mg/g, which is ~2.7% of the mass sorbent. The obtained maximum capacity of a 1 000-mg C18 sorbent for the oil and grease is within the range suggested by Majors (1986) and Van Horne (1990), *i.e.,* 1–5% of the sorbent mass. The extraction efficiency decreases when the maximum capacity of the sorbent has been exceeded, allowing material to pass through the column. The extraction efficiency was not analyzed statistically (*i.e., t*-test) as only duplicate samples were performed on each sample volume.

Based on the results shown in Figure 3, it is concluded that a minimum sample volume of 500 mL is needed for the oil and grease analysis using the developed C18 SPE method. A sample volume of <500 mL may cause inefficiency of extraction that will lead to false low oil and grease results (as shown by the low recovery of oil and grease at 250 mL sample volume in Figure 3). Similarly, large sample volume such as 2 000 mL should be avoided as it may cause the breakthrough of oil and grease compounds from the C18 column. Figure 3 also shows that 500–1 500 mL is the range of sample volume that is suitable for a 1 000-mg size C18 SPE column. The sample size may need to be adjusted as a function of the expected concentration.

**Matrix interferences.** In addition to the deionized water, a known amount of motor oil solution also was spiked into environmental samples that were collected from a storm drain. To avoid the clogging of the C18 column, the environmental samples were filtered with a 1-$\mu$m glass fiber filter paper before addition of the known oil and grease solution. The C18 SPE conditions used were 1 000-mg C18 column, 500 mL of sample volume, 5% of isopropanol for sample pretreatment, and the E4 elution scheme. The average percentage recovery of oil and grease from these environmental spiked samples then was compared with those obtained from the synthetically spiked samples (Table 3). The obtained results show that percentage recoveries of oil and grease in the environmental spiked samples were almost the same as those obtained from the synthetic samples. There was no significant difference, at the confidence level of 0.10, between these two types of samples. This shows that the developed C18 SPE procedure can be used in environmental samples with a complex mixture of compounds.

**Comparison of C18 SPE with LLE.** The extraction efficiency of the C18 SPE was compared with the conventional liquid-liquid extraction for the oil and grease analysis. The obtained results, as shown in Table 4, show that the extraction efficiency of C18 SPE is greater than LLE. The average percentage recovery of oil and grease is 85 and 76% for C18 SPE and LLE, respectively. The extraction efficiency of the C18 SPE is also significantly different than the LLE at the confidence interval of $\alpha = 0.10$. This shows that the developed C18 SPE procedure is a good candidate for replacing LLE for oil and grease analysis.

In addition to the improved and more consistent recovery of oil and grease, the C18 SPE was also able to recover more volatile components of oil and grease than the conventional LLE. This should be anticipated because there is much less solvent to evaporate. The C18 SPE and LLE extracts obtained from an aqueous sample spiked with crude oil were analyzed with GC-FID. Unlike used motor oil, crude oil contains many low-molecular-weight hydrocarbons. The obtained chromatograms of the LLE and SPE extracts are shown in Figures 4 and 5, respectively. From Figure 4, it was observed that there were no peaks detected between the retention time of 7.5 and 13 min, whereas numerous peaks were detected in the SPE extract (Figure 5). The most volatile compounds are not recovered by either method (see Figure 6). This shows that some of the semivolatile components of the oil and grease were lost during the LLE process.

Several stormwater runoff samples also were analyzed for oil and grease using the abovementioned C18 SPE and LLE methods, and the results are shown in Table 5. The oil and grease results using LLE method was found to be lower than those obtained from the C18 SPE method. These results support the results presented earlier in this paper that show the SPE procedure is capable of accurately quantifying oil and grease in a complex mixture, such as normally found in environmental samples.

**Comparison to commercially available procedures.** The extraction efficiency of the C18 SPE was also compared with those obtained from using the EnvirElut™ Oil and Grease column (Varian) and Empore™ Oil and Grease disk (3M). The extraction procedures recommended by the manufacturer were used for these two methods. Table 6 shows the percentage recoveries of oil and grease using the proposed C18 SPE procedure as well as two commercial procedures.

**Table 4—Comparison of percentage recovery of C18 SPE and LLE (synthetic samples).**

| Analytical method | Number of analyses | Mean ± SD | CL ($\alpha$ = 0.10) |
|---|---|---|---|
| C18 SPE | 4 | 85 ± 2 | 84-86 |
| LLE | 4 | 76 ± 4 | 73-79 |

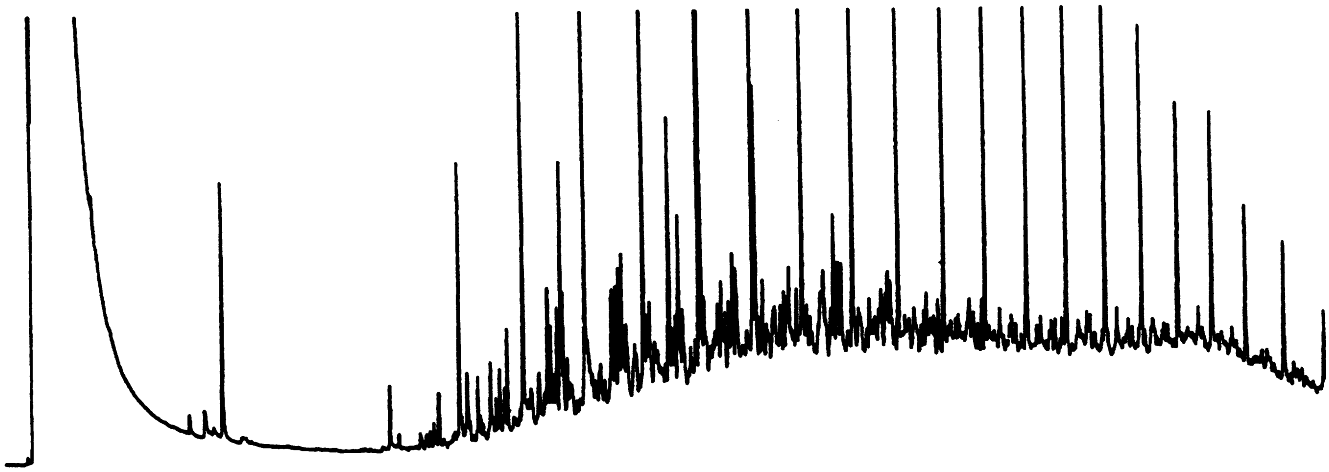

Figure 4—Gas chromatogram of LLE extract of a crude oil sample at threefold dilution.

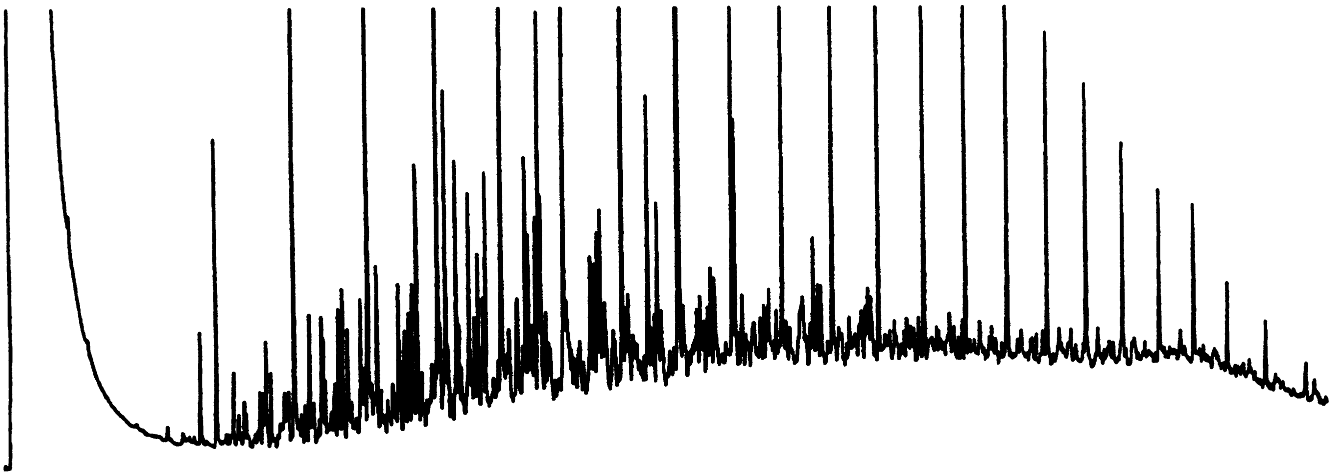

Figure 5—Gas chromatogram of C18 SPE extract of a crude oil sample at threefold dilution.

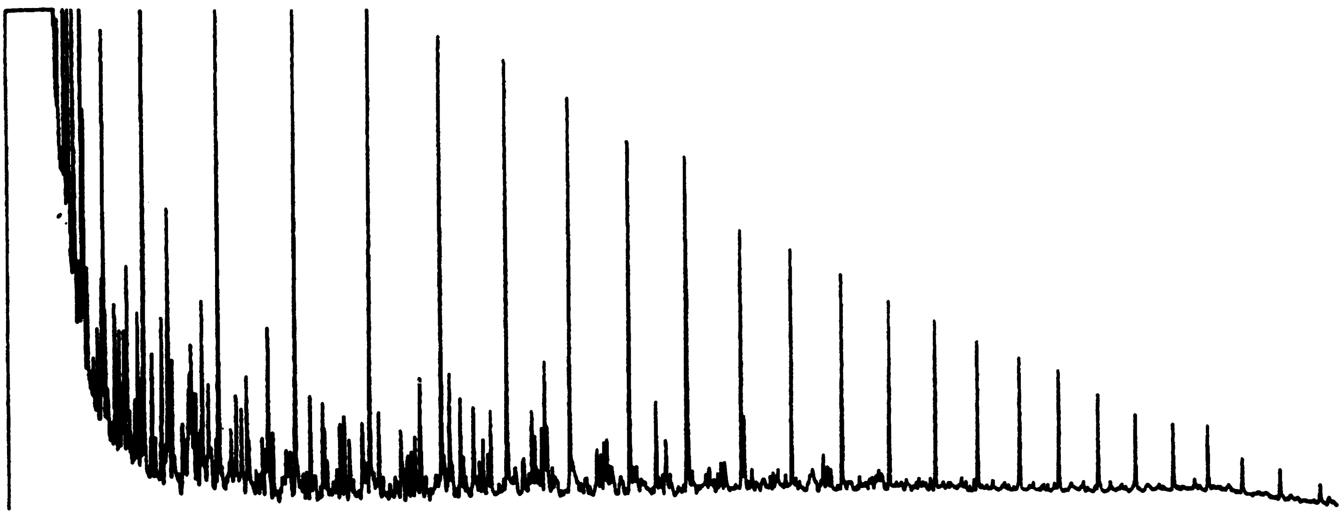

Figure 6—Gas chromatogram of an unextracted crude oil sample at threefold dilution.

**Table 5—Oil and grease results of several stormwater runoff samples using C18 SPE and LLE methods.**

| Sample | Type of sample | Oil and grease, mg/L | |
|---|---|---|---|
| | | C18 SPE | LLE |
| 1 | Stormdrain sample | 2.43 | 1.96 |
| 2 | Stormdrain sample | 8.63 | 7.23 |
| 3 | Stormdrain sample | 30.11 | 24.61 |
| 4 | Runoff from a parking facility | 17.17 | 16.19 |
| 5 | Runoff from a parking facility | 13.98 | 8.39 |
| 6 | Runoff from a parking facility | 9.31 | 4.07 |

Preliminary studies showed low recoveries of oil and grease using the procedures recommended by the manufacturer for the EnvirElut™ Oil and Grease column, where an average of 71% of oil and grease was recovered. When 5% (v/v) of IPA was used instead of the 1% (v/v) as suggested by the manufacturer, a dramatic improvement of the percentage recovery from 71 to 89% was observed (see Table 6). Based on four replicate extractions, it was found that there is no significant difference (at $\alpha = 0.10$) between the C18 SPE column and the EnvirElut™ Oil and Grease column with 5% (v/v) IPA for oil and grease analysis. The proposed C18 SPE procedure is comparable to EnvirElut™ Oil and Grease column for the oil and grease analysis. The EnvirElut™ Oil and Grease columns are approximately twice as expensive as the C18 SPE columns. The EnvirElut™ Oil and Grease procedure also uses 10 mL more solvent than the proposed C18 SPE method.

Based on the procedures recommended by the manufacturer, preliminary studies of Empore™ disk recovered <70% of oil and grease. The percentage recovery of oil and grease improved only slightly after 5% (v/v) of IPA was added into the sample before extraction. Sample flow rate through the disk also was used to enhance recovery; unfortunately, recovery was the same at reduced flow rate. The average percentage recovery of oil and grease was 74%, which was significantly lower than the recoveries of both the C18 SPE and EnvirElut™ Oil and Grease columns.

**Dissolved oil and grease versus total oil and grease.** Oil and grease is often separated into two classes: "free" and "dissolved." Free oil and grease refers to the oil and grease floating on the surface of the water or adsorbed to the container walls. Dissolved oil and grease refers to that portion that is truly dissolved and colloidal particles that are so small that they cannot be removed by flotation or sedimentation. The previously described analysis concentrated mainly on the total extractable oil and grease, *i.e.,* the combination of free and dissolved oil and grease. To show that the proposed C18 SPE procedure is capable of detecting both free and total oil and grease, a modified protocol was developed. A sample was prepared in the normal way except that it was allowed to sit, undisturbed, for 24 h. In this way, the free oil and grease floated to the surface or adsorbed to the container walls. The polytetrafluoroethylene tubing used to transfer the solution from the sample container to the C18 column was submerged halfway below the surface of the sample. Isopropanol was not added into the sample before introduction to the C18 column. Only the half sample was pumped through the SPE column. In this way, no oil and grease that was adsorbed to the container walls or floating on the liquid surface was analyzed. The obtained eluate was used to calculate the concentration of "dissolved oil and grease" using Equation 1. A range of total extractable oil and grease concentration from 6 to 320 mg/L was studied. Figure 7 shows the relationship between the total extractable and dissolved oil and grease. The results show that as the total extractable oil and grease concentration increases, the dissolved oil and grease concentration also increases. However, at ~220 mg/L of total extractable oil and grease, the dissolved oil and grease concentration saturates and remains almost unchanged. This information suggests that all containers and tubing, not made of polytetrafluoroethylene, which contact the sample during the analysis, should be washed with solvent to avoid sample bias by adsorption. Using this technique with the proposed C18 procedure recovers total oil and grease with ~90% recovery.

This procedure may require that the sample be filtered before analysis. Fine suspended solids may clog the SPE column. The conventional LLE procedure does not require filtration before analysis. The suspended solids will be extracted partially in the LLE procedure. When the oil and grease concentration adsorbed to suspended solids is desired, the soxhlet extraction (APHA, 1995) is recommended. The LLE procedure may not completely extract the suspended solids, and the SPE procedure may suffer from clogging columns.

**Conclusions.** The C18 SPE procedures developed in this study showed excellent potential for oil and grease analysis. Greater recovery of oil and grease was observed using the C18 SPE procedures as compared with the conventional liquid-liquid extraction, and its efficiency is also comparable with those obtained from the EnvirElut™ Oil and Grease column. The loss of some semivolatile components of the oil and grease can also be prevented when the SPE procedure is used. The volume of solvent was reduced and more reproducible results were obtained using the C18 SPE method as compared with liquid-liquid extraction. The C18 SPE procedures require an average of 2 h per 500 mL sample analysis, which is approximately the same as liquid-liquid extraction. The length of analysis time might be reduced using a higher flow rate (*i.e.,* >5 mL/min). Analysis in parallel using multiple head pumps will reduce the analysis time for multiple samples.

The proposed procedure was designed to analyze the soluble oil and grease in the stormwater runoff samples. When using the proposed procedure it is recommended that the effects of

**Table 6—Percentage recoveries of various extraction methods.**

| Method | Mean ± SD[a] | CL ($\alpha = 0.10$) |
|---|---|---|
| C18 SPE | 88 ± 2 | 86–90 |
| EnvirElut™ Oil and Grease (1% IPA) | 71 ± 3 | 69–73 |
| EnvirElut™ Oil and Grease (5% IPA) | 89 ± 5 | 85–93 |
| Empore™ Disk | 74 ± 6 | 71–77 |

[a] The results of C18 SPE and EnvirElute™ Oil and Grease columns were based on four replicate extractions. The results of the Empore™ disk were based on 14 extractions.

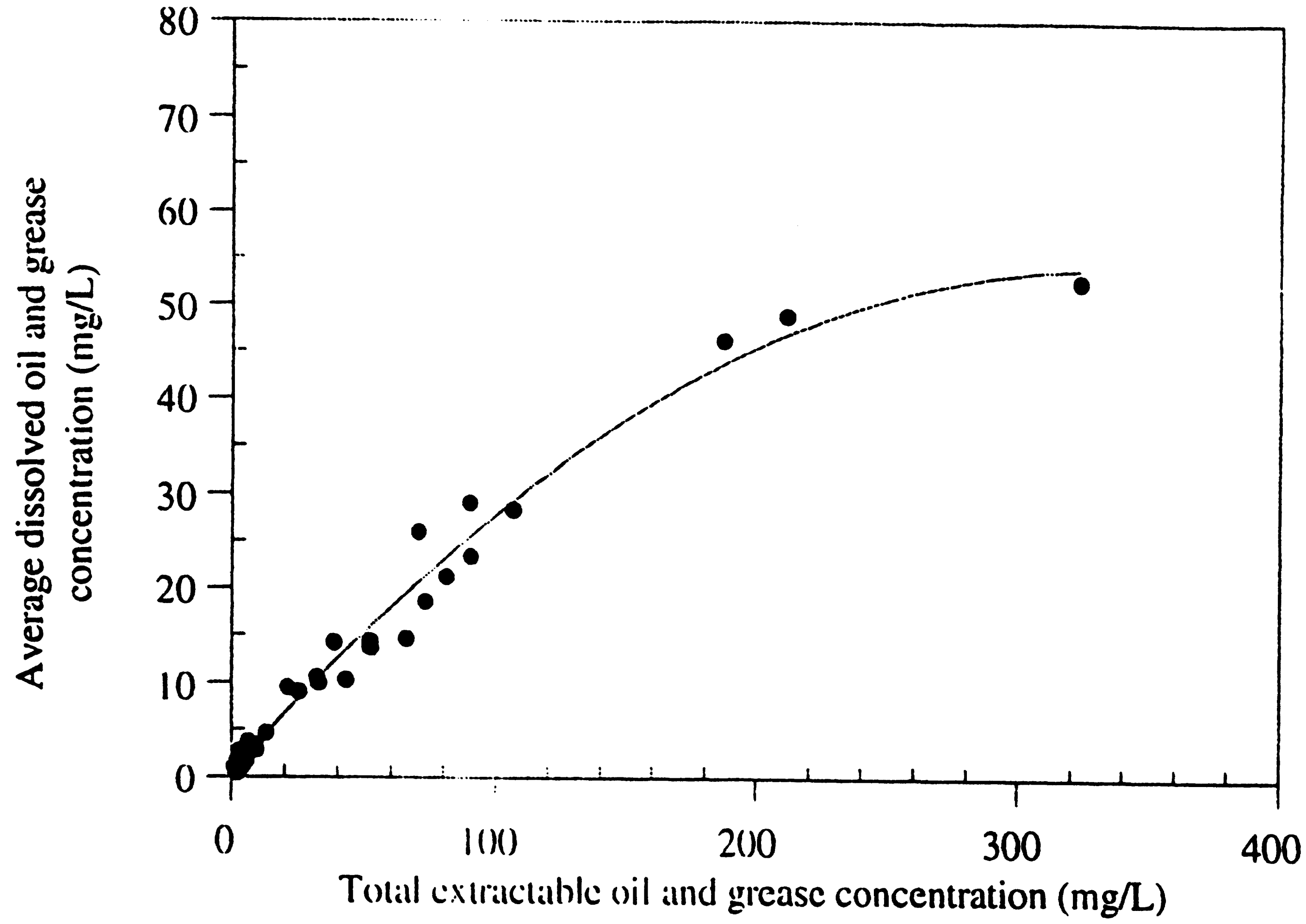

**Figure 7—Correlation of total extractable oil and grease and dissolved oil and grease.**

several variables (such as sample volume and isopropanol volume) be considered. For example, 25 mL isopropanol was optimal in this research, but may be different for different sample types (*e.g.*, high ionic strength samples or industrial wastewater). The proposed procedure also has advantages when fractionation or analysis of the extracted oil and grease is required. The reduced analyte volume means that the extracts are more concentrated, which facilitates analysis using gas or liquid chromatography or gas chromatography/mass spectrometry.

## Acknowledgments

**Credits.** This work was supported in part by contracts from the Los Angeles Department of Water and Power, and the Santa Monica Bay Restoration Project.

*Submitted for publication June 9, 1995; revised manuscript submitted October 24, 1995; accepted for publication November 21, 1995..*

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## References

Carter, W. P. L. (1994) Development of Ozone Reactivity Scales for Volatile Organic Compounds. *J. Air Waste Manage. Assoc.*, **44**, 881.

Chladek, E., and Marano, R. S. (1984) Use of Bonded Phase Silica Sorbents for the Sampling of Priority Pollutants in Wastewaters. *J. Chrom. Sci.*, **22**, 313.

Kao, A. S. (1994) Formation and Removal Reactions of Hazardous Air Pollutants. *J. Air Waste Manage. Assoc.*, **44**, 683.

Majors, R. E. (1986) Sample Preparation for HPLC and Gas Chromatography Using Solid-Phase Extraction. *LC-GC*, **4**, 10, 972–984.

McDowall, R. D., Piarce, J. C., Murkitt, G. S. (1986) Liquid-Solid Sample Preparation in Drug Analysis. *J. Pharm. Biomed. Anal.*, **4**, 3.

American Public Health Association (1995) *Standard Methods for the Examination of Water and Wastewater.* Washington, D.C.

Stenstrom, M. K., Fam, S., Silverman, G. S. (1986) Analytical Methods for Quantitative and Qualitative Determination of Hydrocarbons and Oil and Grease in Water and Wastewater. *Environ. Technol. Lett.*, **7**, 625.

Stenstrom, M. K., Silverman, G. S., Bursztynsky, T. A. (1984). Oil and Grease in Urban Stormwaters. *J. Environ. Eng.*, **110**, 58.

Van Horne, K. C. (Ed.) (1990). Sorbent Extraction Technology. Analytichem International, Inc., Harbor City, Calif.

Wells, M. J. M., and Michael, J. L. (1987) Reverse-Phase Extraction for Aqueous Environmental Sample Preparation in Herbicide Residue Analysis. *J. Chrom. Sci.*, **25**, 345.